

## QUANTITATIVE ULTRAVIOLET ANALYSIS OF $C_{10}$ - $C_{15}$ NAPHTHALENES IN HYDROCARBON OILS

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### INTRODUCTION

The characterization of neutral oils from low-temperature coal tars required the development of a quantitative determination of alkylnaphthalenes according to their degree of substitution.

The near ultraviolet spectrum of naphthalene consists of three principle regions of absorption which, according to Clar's classification (13), are called the  $\beta$ -, para-, and  $\alpha$ -band systems and are related to the three band systems of benzene. The principle bands of these systems are located at 220.7, 275.5, and 311.5 m $\mu$  ( $\log \epsilon$  5.08, 3.77, and 2.40, respectively). For the quantitative analysis of  $C_{10}$ - $C_{11}$  naphthalenes, the use of the  $\alpha$ -bands has been reported by Neimark (31), Adams (2), and Coggeshall (15). An ASTM method (4) for total naphthalenes in jet fuels using the para bands has been adopted and the extent of interference of other aromatics pointed out. Snyder (34) has said that the ASTM procedure using the para bands for naphthalene determination is inapplicable in gasoline samples because of serious interference of monoaromatics. Mixtures of coal tar neutral oils analyzed in this laboratory (23) also prohibits the use of the para bands because of possible interference from other aromatics. Therefore, the present paper describes a method developed for the analysis of  $C_{10}$ - $C_{18}$  alkylnaphthalenes using the most intense  $\beta$ -band of naphthalenes, thereby improving sensitivity and increasing selectivity by reducing interference from other classes.

### EXPERIMENTAL

As part of a recently developed low-temperature coal tar assay (23), total naphthalenes were isolated from a high-quality neutral oil using liquid chromatography on a gas chromatography analog. The detailed procedure for this chromatographic technique has been described (21, 22). For the separation, a weighed quantity close to 1 gram of the high-quality neutral oil was introduced to a 25-ft length of 3/8 in. tubing packed with 80-100 mesh F-20 alumina containing 4 weight-percent water and pretreated with spectral grade cyclohexane. The charge was eluted with cyclohexane under 75 psig nitrogen. Fractions of 14 ml each were collected, and elution of naphthalenes was followed by the automatic recording of a chromatogram with an ultraviolet absorption monitoring device. Ultraviolet spectra were obtained on the cyclohexane solutions from tubes corresponding to the chromatogram peaks, using a Perkin-Elmer 350 ultraviolet spectrophotometer and matched quartz absorption cells.

For the absorptivity data, pure samples of naphthalenes and all methyl- and ethylnaphthalenes  $C_{10}$ - $C_{12}$  were commercially available. Zone refined samples from James Hinton, 358 Chicago Ave., Valparaiso, Fla., were used when possible

and were found to be the highest purity available. Out of the 14 possible isomers of trimethylnaphthalenes, the only commercially available samples were the 1,3,7-, the 2,3,5-, and the 2,3,6- isomers. All samples were weighed on a microbalance for the absorptivity data. A study of the  $\beta$ -band for naphthalene and monomethylnaphthalenes showed that these classes behaved in accordance with the laws of Lambert and Beer and it was assumed that higher homologs also obeyed these laws in the concentrations used.

## RESULTS AND DISCUSSION

Elution Chromatographic Separation. - Naphthalene and its alkyl derivatives emerged from the column relatively free from other classes of compounds, thus allowing the application of ultraviolet quantitative procedures. Aliphatic material, biphenyls, hydroaromatics, and monoaromatics were all eluted before naphthalenes, with only a small amount of overlapping. Three-ring compounds such as dibenzofurans, fluorenes, phenanthrenes, and anthracenes are eluted later and do not interfere seriously. Within naphthalenes as a class, the parameter which causes a pronounced change in adsorbability was the degree of substitution, while position-substitution effects and size-of-alkyl group effects were secondary. Thus, all isomers of mono-, di-, tri-, tetra-, and pentaalkylnaphthalenes were eluted as classes and were sufficiently separated from each other, as evidenced by marked changes in the ultraviolet spectra of successive fractions.

Figure 1 for a synthetic mixture of four pure naphthalenes shows that overlapping occurs more extensively in lower classes and that increased alkyl substitution is effective in enhancing class separation. Klemm (25) points out that methylation of naphthalene enhances adsorbability but increasing the bulkiness of the substituent group decreases adsorbability. Snyder's data on naphthalenes agrees with the increase of adsorbability with alkylation (35), but shows very little difference in adsorbability of 1-ethyl-, 1-n-butyl-, and 1-n-hexylnaphthalene (36). Ethyl groups were known to be present in the naphthalene mixtures analyzed; a detailed characterization of a bituminous coal tar (11) and recent gas chromatographic data on the naphthalene fractions isolated from 18 tars (23) showed that monomethylnaphthalenes comprised 16 percent of the total amount for monosubstituted naphthalenes and that ethylmethylnaphthalenes were 16 percent of the total amount for disubstituted naphthalenes.

Figure 2 shows that the wavelength of the  $\beta$ -band for naphthalenes containing alkyl groups other than methyl depends more on the number than on the size of the substituent. Hence, any marked change in adsorbability of an alkyl naphthalene from that of the correspondingly substituted methylnaphthalene would be readily detected by its  $\beta$ -band. Since such a change was not observed either in the elution of neutral oil mixtures or synthetic mixtures containing ethylnaphthalenes, it was assumed that naphthalenes containing ethyl groups are eluted with methylnaphthalenes with the same degree of substitution. Further, it is assumed that their amounts were accurately accounted for because their absorptivities were not significantly different from methylnaphthalenes with the same degree of substitution. Small differences in adsorbability among some of the various isomers in mono-, di-, and trimethylnaphthalenes were observed in pure compound elution runs.

Figure 3 shows the results for a neutral oil from a low-temperature tar produced from a Colorado subbituminous coal in a fluidized-bed with internal heating at 500° C.

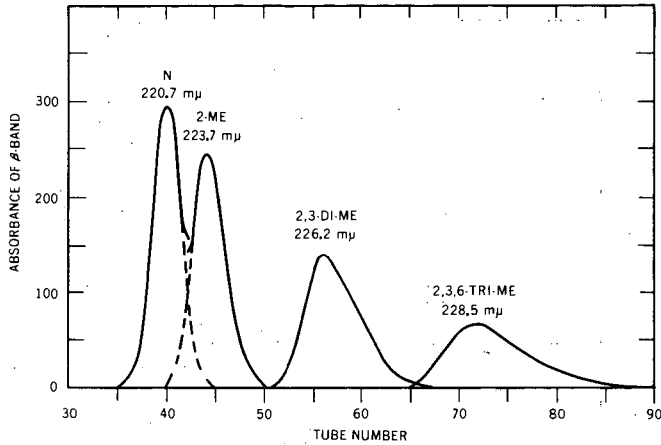


FIGURE 1. - Elution of a Synthetic Mixture of  $C_{10}$ - $C_{13}$  Naphthalenes.

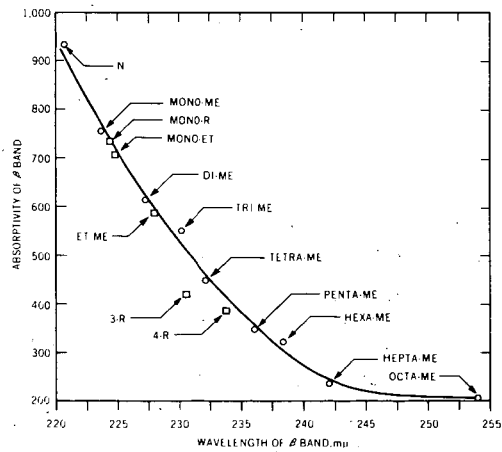


FIGURE 2. - Wavelength and Absorptivity Data for  $C_{10}$ - $C_{18}$  Alkyl naphthalenes.

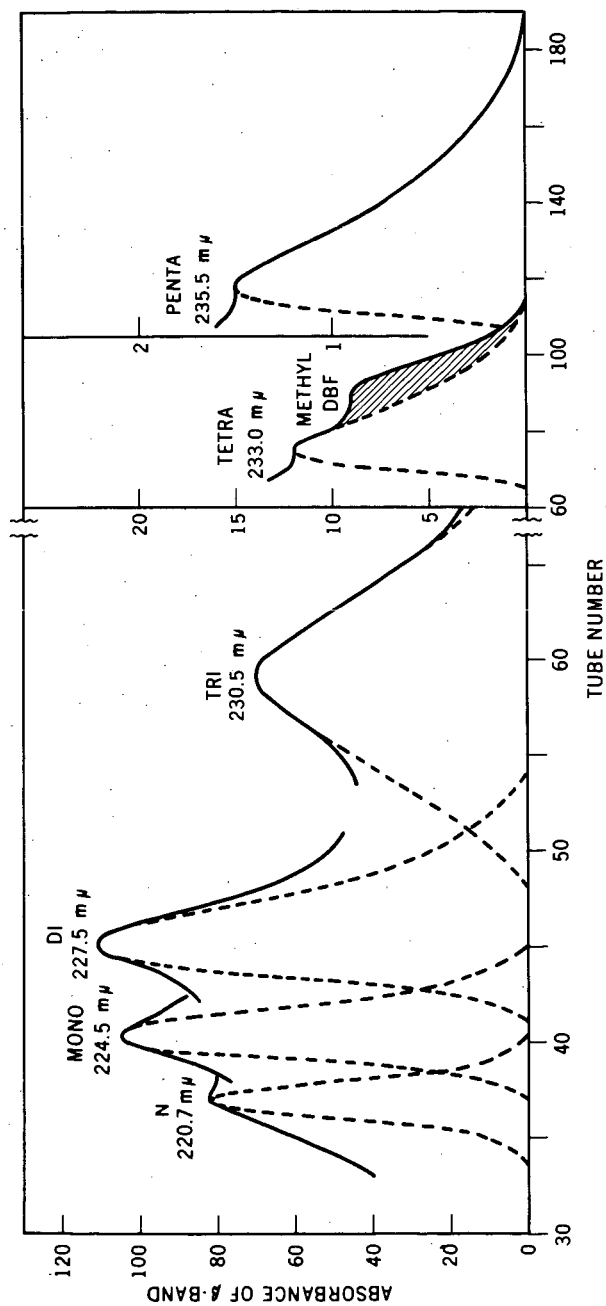


FIGURE 3. - Elution of a Neutral Oil, C<sub>10</sub>-C<sub>15</sub> Naphthalenes.

Since tube number is arbitrary and columns gradually deactivate with extended use, the relative retention of each class to an internal standard can be utilized to aid in the determination of peak tubes, particularly in the broader bands of pentamethylnaphthalenes. For this purpose, 20  $\mu$ l of o-ethyltoluene was added to each sample. Since the synthetic mixture shown in Figure 1 and the neutral oil shown in Figure 3 were run several months apart, the peak tube numbers do not agree. However, their relative retentions are comparable. Figure 1 gives relative retentions of 1.5, 1.7, 2.2, and 2.8 for naphthalene and one isomer each of mono-, di-, and trimethylnaphthalene classes, relative to o-ethyltoluene in tube 26. Figure 3 gives relative retention values of 1.4, 1.5, 1.7, and 2.3, respectively, for natural, complex mixtures of these classes, relative to o-ethyltoluene in tube 26.

**Determination of Ultraviolet Calibration Data.** - It has been observed by Mosby (30) for ultraviolet spectra of  $C_{14}$ - $C_{16}$  methylnaphthalenes that with increasing methylation there are bathochromic shifts relative to naphthalene. Dannenberg (16) has shown that for the  $\beta$ -band of naphthalenes there is a constant bathochromic shift of 3  $m\mu$  per alkyl substituent introduced to the ring, independent of its bulkiness and its position in the ring up to the octa-substituted compound. The data presented in Table 1 for  $C_{10}$ - $C_{18}$  methylnaphthalenes support this wavelength correlation with the exception of octamethylnaphthalene. A study of both wavelengths and absorptivities was made on  $C_{10}$ - $C_{18}$  alkyl naphthalenes, making extensive use of literature data. Ultraviolet spectral data have been reported on all of the 14 possible trimethylnaphthalenes (3, 5, 8, 9, 12, 14, 17, 18, 19, 24, 28, 32, 33), 8 of the tetramethylnaphthalenes (3, 6, 7, 8, 27, 30, 33, 37), 2 pentamethylnaphthalenes (1, 30), 4 hexamethylnaphthalenes (1, 10, 30), 1 heptamethylnaphthalene (1), and octamethylnaphthalene (1, 29). No solvent corrections were applied to the literature data, which are reported both in alcohol and hydrocarbon solvents. Mosby (30) reports that for a tetramethylnaphthalene the only observable difference in methanol and isooctane spectra is a very slight hyperchromic displacement (intensity increase) of the longer wavelength maxima.

As shown in Table 1 and Figure 2 for the  $\beta$ -band there is both a bathochromic and hypochromic displacement (shift to longer wavelength and intensity decrease) as the number of alkyl groups increase in the naphthalene ring. The values of wavelength and absorptivities for the various isomers in each carbon number class are in ranges narrow enough to justify the use of averages. Table 1 presents the arithmetic averages determined for each class of methylnaphthalenes. A plot of carbon number vs. arithmetic average of molar absorptivity yields a linear relationship. However, for application to the present analysis the relationship shown in Figure 2 is shown to have more usefulness. This plot combines both the bathochromic and hypochromic shifts by plotting average absorptivity vs. average wavelength of the  $\beta$ -band for each class of methylnaphthalenes. Values for other alkyl naphthalene classes were added to the plot after the curve for methyl data was drawn. Points shown in squares for monoalkyl-, ethylmethyl-, trialkyl-, and tetraalkylnaphthalenes (where  $R = C_{13}$ - $C_{16}$ ) were obtained from averages of 26 literature spectra.

While the absorptivities and wavelengths for each class fell into definite ranges, it must be pointed out that the  $\beta$ -band is not insensitive to isomerism. A close examination of the data reveals that in addition to the relationship of absorptivity with degree of substitution, it is also subject to position substitution effects.

TABLE 1. - Ultraviolet data for the  $\beta$ -band of  $C_{10}$ - $C_{18}$  naphthalene classes

Class	Source	Average wavelength, $m\mu$	Average absorptivity
Naphthalene	Pure compound	220.7	933
Monomethylnaphthalenes	Pure compounds	223.7	754
Monoethylnaphthalenes	Pure compounds	224.8	709
Dimethylnaphthalenes	Pure compounds	227.3	612
Trimethylnaphthalenes	34 literature values	230.1	550
Tetramethylnaphthalenes	12 literature values	232.1	446
Pentamethylnaphthalenes	2 literature values	236.0	346
Hexamethylnaphthalenes	9 literature values	238.3	321
Heptamethylnaphthalenes	1 literature value	242.0	237
Octamethylnaphthalenes	2 literature values	254	205

TABLE 2. - Ultraviolet data for the  $\beta$ -band of individual  $C_{11}$ - $C_{12}$  naphthalenes

Compound	Wavelength, $m\mu$	Absorptivity
1-Methylnaphthalene	223.7	657
2-Methylnaphthalene	223.7	850
1-Ethylnaphthalene	224.8	609
2-Ethylnaphthalene	224.8	808
1, 2-Dimethylnaphthalene	227.5	544
1, 3-Dimethylnaphthalene	227.8	564
1, 4-Dimethylnaphthalene	227.7	352
1, 5-Dimethylnaphthalene	227.3	571
1, 6-Dimethylnaphthalene	226.9	523
1, 7-Dimethylnaphthalene	227.2	692
1, 8-Dimethylnaphthalene	228.2	572
2, 3-Dimethylnaphthalene	226.2	653
2, 6-Dimethylnaphthalene	226.6	868
2, 7-Dimethylnaphthalene	227.3	785

The  $\beta$ -band of naphthalene has been assigned to longitudinal polarization by Klevens (26). The longitudinal direction is defined as being parallel to the long axis of the molecule while transverse is perpendicular to the long axis of the molecule. Jaffé (20) has discussed the importance of the position of substitution upon the longitudinal and transverse polarizations possible within the naphthalene molecule and the effects on wavelength and intensity. However, he does not discuss these effects with regard to the  $\beta$ -band. An examination of all the classes of naphthalenes shows no correlation of structure with wavelength within each class since the ranges are so narrow. It is to be expected that ring substitution will distort the charge distribution within the naphthalene ring and that this effect will influence the intensity of absorption. The following position substitution effects on the absorptivity of the  $\beta$ -band for four classes of naphthalenes were noted:

(1) For monomethylnaphthalenes, Table 2 shows that substitution in the 1-position causes a greater hypochromic shift relative to the value for naphthalene than 2-substitution.

(2) In the class of dimethylnaphthalenes, Table 2 shows that substitution in the 1,4-position produces the greatest hypochromic shift relative to the value for naphthalene while substitution in the 2,6-position produces the least.

(3) A study of the literature data on trimethylnaphthalenes shows that the 1,4,6-isomer has the lowest absorptivity, thus indicating again a greater hypochromic effect with substitution in the 1 and 4 positions.

(4) Within the class of tetramethylnaphthalenes, the 1,4,6,7-isomer has the lowest absorptivity value (320) while the 2,3,6,7-isomer has the largest (686).

Since, as previously stated, ethyl groups are present in coal tar neutral oils and, specifically, monoethylnaphthalenes, it was thought they should be included in the average absorptivities used for the determination of monoalkylnaphthalenes. An arithmetic average of 731 was obtained for the two classes. Since position substitution has a pronounced effect on absorptivity of monoalkylnaphthalenes, a further check on the validity of this average was made by calculating a weighted absorptivity. Data used in obtaining this weighted absorptivity was based on gas chromatographic analysis of each of these four compounds in the isolated naphthalene fractions from 18 individual neutral oils. A value of 757 was obtained which represents a difference of only 3.4 percent from the arithmetic average. In the same manner as for monoalkylnaphthalenes, a weighted average absorptivity was determined for dimethylnaphthalenes. A value differing from the arithmetic average was expected here since there is not an even distribution of the amounts of the 10 dimethyl- isomers in these neutral oils. For example, 1,8-dimethylnaphthalene has not been identified in any coal tar neutral oil. However, a value of 639 was obtained which differs only 4.2 percent from the arithmetic average of 612 for all 10 isomers. In summary, for analysis of coal tar neutral oils, tailored absorptivities of 757 and 639 were used for the mono- and dialkylnaphthalene classes respectively, and absorptivities obtained from the literature were used for the tri-, tetra-, and pentamethylnaphthalenes.

**Ultraviolet Analysis.** - Throughout successively eluted portions of a neutral oil a regular stepwise bathochromic shift of the  $\beta$ -band occurs and the wavelength of each shift is in good agreement with the wavelength average determined for each class of methylnaphthalenes shown in Table 1. Corresponding to each wavelength shift a peak occurs in the monitor chromatogram. Data from the ultraviolet spectra of individual tubes were used to construct absorbance distribution curves for the  $\beta$ -band of each class of naphthalene, as shown in Figures 1 and 3. The envelope of the ultraviolet absorbance curve is discontinuous, i. e., it is constructed of data from several wavelengths. The plot for a single wavelength was followed as long as that wavelength predominated, i. e., until a combination tube was reached. The absorbance plot was then continued at the new wavelength. In the neutral oil samples analyzed there were consistently six wavelength changes corresponding to  $C_{10}$ - $C_{15}$  alkyl naphthalenes. A graphical method was utilized to resolve the individual absorbance distribution curves for each class. Using qualitative changes exhibited throughout the entire ultraviolet spectrum (naphthalene  $\alpha$ - and para- bands also vary qualitatively with increasing degree of alkylation), it is possible to follow the key tubes representing the first appearance, the peak and the disappearance for each class of naphthalene, even in the presence of overlapping. These tubes and all combination tubes are then marked on the absorbance plot. The absorbance curve is then drawn according to this information. The dashed lines shown in Figures 1 and 3 indicate this deduced data. Knowledge of curve shapes and characteristic tailing was obtained from pure compound runs. After the absorbance curve for an individual class was constructed in this manner, the absorbance values for each tube were read from this resolved plot and the proper absorptivity used to calculate the weight of that class in a single tube according to the following equation:

$$w = \frac{A \times l4}{a}$$

where  $w$  = weight, milligrams;

$l4$  = volume of one tube, ml;

$a$  = absorptivity of the naphthalene class at the  $\beta$ -band;

$A$  = absorbance of the tube at the  $\beta$ -band.

Extensive interference from non-naphthalenic compounds was not encountered. Both ultraviolet and infrared spectra showed that the lower classes of naphthalene were sufficiently separated from the preceding classes of hydroaromatics, biphenyls, and monoaromatics. A small amount of overlapping introduced negligible error since these classes have minimum absorption in the region of the naphthalene  $\beta$ -band and, in addition, have significantly lower absorptivities at their maxima. When there was qualitative evidence of significant amounts of dibenzofurans, fluorenes, phenanthrenes, and anthracenes overlapping the higher naphthalene classes, there was a change in the shape of the distribution curve, as shown in Figure 3 for the tetramethylnaphthalene class. The amount of absorbance due to this interfering compound was graphically subtracted from the absorbance distribution plot, as shown by the shaded area, considering the shape of the naphthalene curve. In the instance of Figure 3, the compound was identified as a methyl dibenzofuran mixture from its ultraviolet bands. Since the method is graphical, it is not necessary to identify the compound to subtract its absorbance. If this graphical approach is prevented by too close an agreement of peak tubes of interfering compound and



naphthalene class, the standard quantitative method of solution of simultaneous equations can be applied. The absorptivities of these more strongly adsorbed compounds at the naphthalene  $\beta$ -band wavelength are minimum since their wavelength maxima are all in the range 250 to 260 m $\mu$ .

Because of the unavailability of pure samples in the C<sub>14</sub>-C<sub>15</sub> naphthalene range, no realistic synthetic mixture could be tested. However, analysis of the relatively simple mixture shown in Figure 1, using absorptivities specific for the compounds present, gave individual errors of not more than 2 percent absolute. Another accuracy test was made on a mixture synthesized to duplicate the percentages of individual C<sub>10</sub>-C<sub>13</sub> naphthalenes in a neutral oil sample. This test mixture, containing 14 naphthalenes, gave an ultraviolet absorbance distribution curve that showed the same class elutions as obtained with neutral oils. Average class absorptivities were used to determine the amounts of each class. Results showed that no naphthalene class was in error more than 5 percent absolute.

The 18 neutral oils analyzed by this method varied in composition from 5 to 25 percent total alkyl naphthalenes. In spite of this composition variation, wavelengths of each class remained remarkably constant. These analytical procedures for coal tar neutral oils should be applicable to similar hydrocarbon oils from other sources, such as petroleum and shale oil.

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